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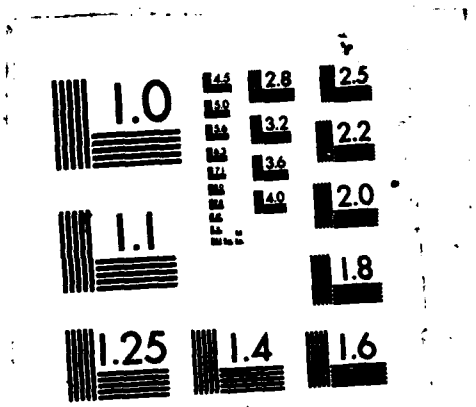

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EVALUATION OF RESINS CURED BY ULTRAVIOLET RADIATION AND IN CONJUNCTION WITH FIBER OPTIC SYSTEMS FOR USE IN THE FIELD REPAIR OF COMPOSITE MATERIALS

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ABSTRACT

Several commercially available resins designed to be cured with ultraviolet radiation were evaluated with respect to their potential for use in the field repair of composite materials. Thermal and mechanical properties of cured resins were evaluated by DSC and TBA techniques. Some cured resins exhibited physical properties which may be suitable for repair applications. None of the tested materials was able to cure when impregnated in woven graphite cloth which strongly attenuates the curing radiation. This prevented the use of fiber optics to cure these systems. KEYWORDS →

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INTRODUCTION

This project is related to an ongoing effort to address the problems associated with the repair of composite materials that are becoming increasingly widespread in military applications. In particular, this project is concerned with field repair of such materials, to be conducted under highly variable conditions with a minimum of support services and equipment. Current repair technology for advanced composite materials involves the use of thermally cured resins and associated equipment to apply heat and pressure as required to fabricate a patch. Low energy curing resin systems, those that cure at ambient temperature or upon exposure to light or other radiation, have attracted interest as alternatives to thermally cured systems now employed. The use of ultraviolet (UV) radiation to cure glass-reinforced composites has been demonstrated,[1] but in the case of graphite- and Kevlar-reinforced materials, the opacity of the reinforcing fiber is an impediment to full exposure of the resin to the curing radiation. It was felt that using optical fiber waveguides, radiation could be introduced within the plies of a composite material, thus effecting a uniform cure throughout an essentially opaque material. This approach has been endorsed by a committee of the National Materials Advisory Board.[2]

MATERIALS

Resins

Ultraviolet curable resin systems are commercially available and are designed principally for use as inks, coatings, and adhesives. This program was initiated without concern for the structural properties of these materials on the grounds that once feasibility of curing resins in a graphite-reinforced composite material had been demonstrated, suitable modifications of existing resins or development of new resins for structural applications could be undertaken.

The commercially available UV curable resins generally fall into one of two categories. The first type consists of acrylates or epoxy resins modified by reaction with acrylic or methacrylic acid to produce acrylate esters which cure by means of a free radical mechanism. The features of these systems are very fast cures (typically seconds for thin layers) and limited thermal stability of the uncured resin. The optimum wavelength radiation to induce the curing reaction is typically 365 nm. The second type of resin is an unmodified cycloaliphatic epoxy resin with a photoinitiator such as $[Ar_2I]^+BF^-_4$ added. These systems exhibit excellent uncured thermal stability (typically stable up to 100°C) and require radiation in the region of 225 nm to effect rapid cure. Sensitizing dyes, such as acridine orange, can be added to change the optimum curing wavelength. These photoinitiators work by generating a so-called superacid, such as HBF_4, in situ when irradiated and cure takes place by a cationic mechanism. Owing to the relatively long lifetime of an ionic species, the cure reaction continues to some extent in these resins after irradiation is stopped.[3] Another feature of the cationic curing mechanism is a sensitivity to organic bases which poison the initiator and prevent cure. As a result, nitrogen containing epoxy resins, such as

1. GILLMAN, H. D., and EICHELBERGER, J. L. *The Application of Ultraviolet Cure Resins for Repair of Composites.* Pennwalt Corp., Contract N62269-77-M7197, Final Report, November 1977, NTIS Accession No. AD-A055731.
2. National Academy Press, *Report of the Committee on Room Temperature Curing Resin Systems,* NMAB-412, March 1984.
3. CRIVELLO, J. V., and LAM, J. H. W. *Diaryliodonium Salts. A New Class of Photoinitiators for Cationic Polymerization.* Macromol., v. 10, no. 6, 1977, p. 1307.

tetraglycidylmethylenedianiline (TGMDA) or diglycidylorthotoluidine [$CH_3(C_6H_4)N(OCH_2CHOCH_2)_2$], cannot be used with cationic initiators. Epoxies based on DGEBA (diglycidyl ether of bisphenol A), such as EPON 828, can be successfully cured by this method.

The resins used with their cure mechanism and approximate compositions are shown in Table 1.

Table 1. ULTRAVIOLET CURABLE RESINS STUDIED

Resin Name (Manufacturer)	Composition	Cure Type
Alcocure EBDMA (Alcolac)	Methacrylic acid ester of DGEBA, 98%.	Free radical
Uniset UV-900 (Amicon)	Urethane acrylate, 40-50% Acrylic monomers, 40-50% Initiators, <5%.	Free radical
Cel-Rad 3600 (Celanese)	Diacrylate ester of bisphenol A.	Free radical
Masterbond UV-10 (Masterbond)	100% UV reactive polymer probably an acrylate.	Free radical
Tactix 742 (Dow)	Triglycidyl ether of tris-(hydroxyphenyl)methane. A trifunctional epoxy resin.	Ionic using FX512*
Cyracure 6110 (Union Carbide)	Cycloaliphatic epoxy resin.	Ionic using FX512*

*Cationic cure initiator, diphenyliodoniumtetrafluoroborate or closely related derivative 60%, with butyrolactone, 40% (manufactured by 3M Company).

Fiber Optics

Several companies produce optical fibers that are transparent to UV radiation, most based on fused silica. Two representative samples were obtained from SpecTran Corporation for use in this study.

EVALUATION

Experiments were conducted to determine the suitability of some of the commercially available UV curing resins for use in graphite-reinforced composite systems. The experiments included determination of the effect of different UV sources on the cure behavior of the neat resins, the effect of increasing resin opacity using carbon black dispersed in the resins, and the behavior of the resin when used with small patches of graphite reinforcing cloth. Both surface irradiation and radiation introduced via optical fibers were used in various experiments.

Epoxy Resins Cured Using Cationic Initiators - Surface Irradiation

A key feature of these resin systems revealed by the experiments is their sensitivity to both the wavelength and intensity of the radiation used. Exposure to a 6500W Xenon Suprasil flash lamp (3 min @ 2 flash/sec, 100 μs/pulse) which did not provide significant UV radiation below 250 nm, was not sufficient to fully cure

a 1-ml sample of resin (Cyracure 6110 + FX512 4%). Similarly, one-hour exposure to 2-5W vacuum UV (Pen-Ray) Hg lamp did not cure the mixture. Exposure to a 100W (33W/in.) high pressure Hg lamp for 40 minutes cured the mixture to a leathery consistency. A 550W (122W/in.) high pressure Hg lamp was able to completely cure a 1-ml sample in 5 to 10 minutes (including approximately 5 minutes warm-up time). Cure times on the order of seconds are reported by the manufacturers using a 200W/in. medium pressure Hg lamp and thin films (inks, coatings) of the resin formulation. In view of the reported tendency of cationically cured resins to continue curing after irradiation was stopped,[3] it was anticipated that the cationic cure system would be most suitable for use in graphite-reinforced composites. In these systems, the cure might be expected to propagate into areas receiving little or no direct irradiation. Repeated experiments showed that this is not the case. In experiments where a neat sample of a resin formulation was partially masked, no cure occurred in the unexposed fraction of the sample while the exposed fraction cured completely. In experiments where a patch of graphite cloth saturated with resin was irradiated, cure took place only at the irradiated surface. There was no evidence of propagation of the cure through the patch. Based on the sensitivity of the cationic curing resins to the intensity of the incident radiation, the possibility that the concentration of active (initiator) species was critical to cure behavior was investigated. Epoxy formulations containing 25% and 50% cationic initiator by weight were prepared and cured in pans that were partially masked from the UV lamp (100W or 33W/in. Hg lamp). No cure into the unirradiated portion of the sample was observed in any case, indicating that either lack of molecular mobility due to crosslinking or termination reactions dominate the system. This observation precludes the possibility of extensive propagation of cure away from the radiation source. Experiments done to determine if the presence of elemental carbon affected the cure showed that low concentrations (approximately 10% w/w) did not prevent cure and such formulations are specified for use as black lithography inks by the manufacturer of the curing agent. Formulations of 50% carbon black (a thick paste) with the resin would not cure. This resin/filler ratio approximates that found in fiber-reinforced composites.

Resins Cured By Free Radical Mechanism - Surface Irradiation

These materials are much less sensitive to the wavelength and intensity of the incident radiation than the cationically cured resins. A 2-5W Pen-Ray Hg lamp was not capable of fully curing a neat 1-2 ml sample of resin, but a 100W (33W/in.) Hg lamp fully cured such a sample with a few minutes exposure (Masterbond UV-10 resin). These resins can also be cured using a 6500W Xenon flash lamp (Germasil or Suprasil). Exposure of 15 seconds @ 2 flash/second (100 μs/pulse) was sufficient to completely cure small samples of neat resin. A sample of resin was also cured after exposure to strong sunlight through a glass window for two days. No further curing takes place in partially cured samples without irradiation. Experiments in which samples were partially masked showed that no cure occurred in unexposed areas of the resins. Inclusion of small quantities (approximately 10% wt) of carbon black did not prevent curing, but when patches of graphite cloth were saturated with resin and irradiated, curing occurred only at the exposed surface.

Thermal and Mechanical Analysis of Cured Resins

Preliminary efforts to characterize the cured resins have employed differential scanning calorimetry (DSC) and torsional braid analysis (TBA) techniques. For Masterbond UV-10, DSC analysis of photocured samples showed that cure was 96% complete after 15 seconds exposure to 2 flash/second of a 6500W Xenon Suprasil flash lamp. After 120 seconds exposure to the same radiation, the cure was 99% complete. A broad T_g centered around 76°C was observed for both samples. TBA analysis of a sample of MB UV-10 cured on a glass braid using a 100W (33W/in) Hg lamp for one hour showed the onset of softening at 25°C. This method also showed that even after this extensive irradiation period, ultimate T_g is not achieved for samples cured on glass braids. Heating the sample through 200°C caused the softening onset temperature to rise about 35°C. The observation of low softening temperatures may reflect the fact that the resins being tested are formulated mainly for use as coatings and as such require some flexibility at ambient temperatures. Table 2 shows the results of TBA analysis of all the resins in the test. Several of these materials exhibited softening temperatures adequate for many types of service, supporting the idea that photocurable resin systems can be made viable for repair applications. If a very high ultimate T_g is critical, it may be necessary to use heat in conjunction with another curing mechanism (such as UV initiation) or to revert to thermally induced cure altogether.

Table 2. TBA ANALYSIS OF ULTRAVIOLET CURABLE RESINS

Resin	Scan	Onset of Softening (°C)
Alcocure EBDMA	1	77
	2	77
Uniset UV-900	1	0
	2	0
Cel-Rad 3600	1	0
	2	0
Masterbond UV-10	1	25
	2	35
Tactix 742	1	48
+ 4% FX512	2	77
	3	114
	4	132
Cyracure 6110	1	86
+ 6.9% FX512	2	86

TBA conditions: 1.5 °C/minute from -40 through 200°C.
All samples cured on a glass braid using a 100W (33W/in.) medium pressure Hg lamp for 30-60 minutes.
Maximum temperature reached during cure was 60°C.

It should be noted that the long irradiation times used to cure the glass braid composites for the TBA experiment are not typical of these resins, and were not necessary to cure neat samples of the resins or flat glass-reinforced test patches. The reasons for this observation are not fully understood, but it may be that the very small cross section that the TBA braid presents to the UV source results in inadequate radiation flux for rapid cure.

Fiber Optics

No conclusions can be drawn at this time about the suitability of optical fibers for curing composite materials or photoinitiated resins in general. It was not possible to get sufficient energy into a fiber to effect cure at a fiber end with the equipment available. It is necessary to gather and focus diffuse light from a source into an intense spot on the fiber end. Special UV transparent optics are needed to avoid attenuation of the desired wavelengths. These optics were unavailable to us. It is most probable that neat photocurable resins can be cured using optical fibers since high energy transmission applications, such as laser steel cutting, have been demonstrated with these fibers. The unanswered questions concern the nature of the cure at a fiber end, specifically the distance cure can propagate from a fiber end and the shape of the irradiated/cured area. In the absence of resins that can propagate the cure reaction through an opaque material, the need to answer these questions is not acute.

The recent introduction of fiber optics transparent to infrared radiation is a development that may eliminate the requirement that a resin propagates cure away from the radiation source. Infrared radiation could potentially be introduced via optical fibers into a composite patch to initiate a conventional thermal cure. The principal advantage of this approach (as opposed to surface heating) is the ability to heat the patch uniformly and quickly. Efforts are now underway to investigate the potential of this method.

CONCLUSIONS

Current light curing resin technology is not applicable to graphite-reinforced composite materials. This conclusion can almost certainly be extended to include all reinforcing fibers/materials that significantly reduce transmission of the radiation used to induce cure in the material. In the case of the epoxy resins cured by cationic mechanism, it is not completely clear why the curing reaction will not propagate from an irradiated portion of a sample into a portion that has not been directly exposed. One likely possibility is that loss of molecular mobility as cure advances severely limits the extent of such an auto-cure process by preventing migration of monomer to the reactive sites in the growing polymer matrix. Another possibility is that the commercial resins contain impurities that terminate the reaction. In any event, it is considered unlikely that minor changes in formulation or cure conditions will render current photocuring technology suitable for use with composite materials using opaque reinforcing media.

FUTURE RESEARCH

Although currently available commercial UV curable resins are not suitable for use with graphite-reinforced composite materials, it may be possible to design a resin that would work in such an application. By incorporating into a resin a component that liberates a large quantity of heat, for example, it might be possible to photoinitiate fast thermal cure in epoxy resins. Another more exotic approach might be to incorporate a material that undergoes thermal decomposition with fluorescence and can effect an internal photocure.

Further study of the mode of propagation of cure through a resin may lead to suggestions for reactive additives which can propagate a photochemically induced cure reaction from irradiated to unirradiated regions in a resin, or through on opaque, but porous, material saturated with resin.

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DISCLAIMER

The identification of commercial materials by name should not be considered as an endorsement of these products by the United States Government. It is done only for purposes of materials description. In addition, the findings reported herein reflect only on these studies and have no bearing on the adequacy of these materials for their intended uses.

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Key Words

UV curing
Fiber optics
Low energy curing

Several commercially available resins designed to be cured with ultraviolet radiation were evaluated with respect to their potential for use in the field repair of composite materials. Thermal and mechanical properties of cured resins were evaluated by DSC and TBA techniques. Some cured resins exhibited physical properties which may be suitable for repair applications. None of the tested materials was able to cure when impregnated in woven graphite cloth which strongly attenuates the curing radiation. This prevented the use of fiber optics to cure these systems.

U.S. Army Materials Technology Laboratory,
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